Material Robustness Testing and Nondestructive Evaluation Methodology Assessment for Liquid Rocket Engine Composite Nozzles

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Thrust-to-weight ratio requirements for Reusable Launch Vehicle (RLV) main propulsion systems are substantially increased relative to current high performance liquid rocket engines. One approach to achieve these requirements as described in the Access to Space Study is to develop lightweight components. High-temperature composite nozzles offer significant potential for weight reduction and cost savings relative to conventional regeneratively cooled metallic nozzles. The Materials and Processes Laboratory initiated a project to address critical technologies required for the development of high-temperature composite nozzle extensions. The effort is supported by the Long-Term/High-Payoff technologies project of the RLV program. Four technology areas were originally identified: Material systems, nondestructive evaluation (NDE), thermostructural analysis code development and verification, and nozzle attachment methodology. Discussions were held with Government and industry organizations to select areas in which up-front studies would benefit development of a composite nozzle extension supporting RLV concepts. Top priority was given to material systems screening and NDE methodology assessment and tasks were defined for each area. Attachment methodology was judged to be design-specific and not recommended for generic development. It was also determined that resources applied to analytical code development and verification would be most beneficial after final material system selection.

The objective of the materials screening task is to conduct a robustness evaluation of composite material systems, including substrates and coatings, which are candidates for RLV nozzle applications for the purpose of assessing performance capability in simulated rocket nozzle environments. An industry/Government team composed of representatives from Rocketdyne, Aerojet, Pratt and Whitney, Southern Research Institute, MSFC, and LeRC was formed to define and conduct the robustness testing project. The initial step was the identification of critical parameters for material selection including constituents, processes, reinforcement architectures, and oxidation protection mechanisms. The team then defined preliminary requirements for the operational environment and established the types of tests to be used to assess performance capability for mission requirements. Preliminary requirements were established using the Space Shuttle Main Engine combustion gas environment as a baseline; operating temperatures in the range of 2,800 to 3,100 °F; and a 20-mission lifetime, with a 500-sec duration for each mission. Tests include baseline tensile, stressed oxidation, oxidative fatigue with thermal cycling, and thermal expansion. The elevated temperatures (two will be selected) and stress levels for the stressed oxidation and oxidative fatigue tests will be defined based on preliminary design and operational requirements analyses. Preliminary material system selections were made including potential suppliers recommendedd by industry team members. Critiques of team material selections were

Table 7.—*Nozzle material selections for robustness testing.*

Item / Material	Manufacturer	Yarn	Yarn Ht.	Fiber Arch.	Yarn Ends	Interface Coating	Matrix	Densification	Oxide Inhib.	Coating
1.Enhanced C-SiC	Dupont Lanxide	1K T–300	1,700 °C	2D Plain	19/in	PyC Alpha-3	SiC	Isothermal	Yes	CVIP
2.STD C-SiC	Dupont Lanxide	1K T–300	None	2D Plain	19/in	PyC Alpha-3	SiC	Isothernal	No	CVIP
3.C-SiC	BFG	1K T–300	1,800 °C	2D Plain	19/in	PyC	SiC	Isothermal	Yes	CVD SiC
4.CTD C-C	CCAT	1K T–300	1,700 °C	2D Plain	30/in	PyC	C	Phen. Impreg.	No	Pack SiC
5.Hi NIC SiC	Dupont Lanxide	1K Hi Nicalon	None	5 HS	17/in	PyC Alpha-3	SiC	Isothermal	Yes	None
6.Hi NIC SiNC	Dow	0.5K Hi Nicalon	None	2D Plain	16/in	Prop Non C	SiNC	PIP	"Yes"	None
7.Hi NIC–C	BFG	0.5K Hi Nicalon	None	2D Plain	16/in	PyC	C	Isothermal	No	SiC
8.CTD C-C	CCAT/Amercon	1K T-300	1,700 °C	2D Plain	30/in	PyC	C	Phen. Impreg.	No	Si_3N_4

sought from the suppliers as well as recommendations of their “best” materials to meet the requirements. Recommendations were also received from experts at the Air Force Materials Laboratory at Wright Patterson Air Force Base. The basic material systems selected were carbon matrix and ceramic matrix composites reinforced with either carbon fiber (T-300) or silicon carbide fiber (Hi-Nicalon). Detailed descriptions of the particular variations and the material supplier are provided in table 7. Orders were placed with suppliers in June 1996. Tests will be conducted at Southern Research Institute and LeRC.

The second critical technology to enable the use of composite material systems for nozzle extension applications is the capability to nondestructively inspect the components. The objectives of the NDE methodology assessment task are to evaluate and subsequently downselect viable NDE techniques for selected composite material systems. An industry/Government team of NDE specialists from Aerojet, Pratt & Whitney, Rocketdyne, Southern Research Institute, MSFC, and LeRC was formed to define and conduct the project. Discussions were held to identify the concerns and needs of the RLV main propulsion system prime contractors and to determine the capabilities of the suppliers. Based upon this information and input provided from the composite nozzle material systems robustness testing team, standard specimens were designed containing prescribed flaws representative of naturally occurring and critical defects. These are described in table 8 for the two material types, coated carbon-carbon and coated carbon-silicon carbide. The specimens were ordered and will be produced with those manufactured for the materials robustness testing task. Each of the test articles will be inspected at MSFC and LeRC and will be available for inspection by all industry team members. Appropriate NDE techniques in existence at each facility will be evaluated as well as selected promising new techniques available within the NDE community. Detection capabilities and limitations data base for these specimens will be established. Based upon evaluation of detection capabilities, applicability to subscale and full-scale composite nozzles, cycle time, and cost, downselection of NDE techniques will be made. Reference specimens will be available for future NDE calibrations.

TABLE 8.—*Defect test articles and materials for NDE methodology assessment task.*

Defect Type	Defect Description	Defect Level	Coated C-C	Coated C-SiC
I.	Density Variation	Nominal Less 15% Less 30%	2×(2×8 in) 2×(2×8 in) 2×(2×8 in)	2×(1.3×8 in) 2×(1.3×8 in) 2×(1.3×8 in)
II.	Interfacial Bonding	Optimized Less Than Opt. Poor Bonding	2×(2×8 in) 2×(2×8 in) 2×(2×8 in)	– – –
III.	Simulated Delams	Best Effort 2.0 mil Thick Strips 5.0 mil Thick Strips	2×(2×8 in) see II a 2×(2×8 in) 2×(2×8 in)	2×(1.3×8 in) see II a 4×8 2×TH. see III b
IV.	Distorted Plies	Nominal 15° 30°	2×(2×8 in) see II a 2×(2×8 in) 2×(2×8 in)	– – –
V.	Inherent	As Sorted	2×(2×8 in)	2×(4×4in)
VI.	Machined	TBD/ASTM	2×(2×8 in)	2×(4×4in)

Results from both tasks will be summarized in the *1997 MSFC Research and Technology* report.

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